

FORM-PTO-1390 (Rev. 9-2001)		U.S. DEPARTMENT OF COMMERCE PATENT AND TRADEMARK OFFICE		ATTORNEY'S DOCKET NUMBER
TRANSMITTAL LETTER TO THE UNITED STATES DESIGNATED/ELECTED OFFICE (DO/EO/US) CONCERNING A FILING UNDER 35 U.S.C. 371				022701-977
INTERNATIONAL APPLICATION NO. PCT/FR00/01809		INTERNATIONAL FILING DATE 28 June 2000		U S APPLICATION NO (If known, see 37 C F R 1 5) Unassigned 10/018814
				PRIORITY DATE CLAIMED 29 June 1999
TITLE OF INVENTION METHOD FOR MAKING ADIPIC ACID				
APPLICANT(S) FOR DO/EO/US FELIX, Albert and ROQUES, Yves				
Applicant herewith submits to the United States Designated/Elected Office (DO/EO/US) the following items and other information:				
<ol style="list-style-type: none"> <input checked="" type="checkbox"/> This is a FIRST submission of items concerning a filing under 35 U.S.C. 371. <input type="checkbox"/> This is a SECOND or SUBSEQUENT submission of items concerning a filing under 35 U.S.C. 371. <input checked="" type="checkbox"/> This is an express request to begin national examination procedures (35 U.S.C. 371(f)). The submission must include items (5), (6), (9) and (21) indicated below. <input checked="" type="checkbox"/> The US has been elected by the expiration of 19 months from the priority date (Article 31). <input checked="" type="checkbox"/> A copy of the International Application as filed (35 U.S.C. 371(c)(2)) <ol style="list-style-type: none"> a. <input checked="" type="checkbox"/> is attached hereto (required only if not communicated by the International Bureau). b. <input checked="" type="checkbox"/> has been communicated by the International Bureau. c. <input type="checkbox"/> is not required, as the application was filed in the United States Receiving Office (RO/US). <input checked="" type="checkbox"/> An English language translation of the International Application as filed (35 U.S.C. 371(c)(2)) <ol style="list-style-type: none"> a. <input checked="" type="checkbox"/> is attached hereto. b. <input type="checkbox"/> has been previously submitted under 35 U.S.C. 154(d)(4). <input checked="" type="checkbox"/> Amendments to the claims of the International Application under PCT Article 19 (35 U.S.C. 371(c)(3)) <ol style="list-style-type: none"> a. <input type="checkbox"/> are attached hereto (required only if not communicated by the International Bureau). b. <input type="checkbox"/> have been communicated by the International Bureau. c. <input type="checkbox"/> have not been made; however, the time limit for making such amendments has NOT expired. d. <input checked="" type="checkbox"/> have not been made and will not be made. <input type="checkbox"/> An English language translation of the amendments to the claims under PCT Article 19 (35 U.S.C. 371(c)(3)). <input type="checkbox"/> An oath or declaration of the inventor(s) (35 U.S.C. 371(c)(4)). <input type="checkbox"/> An English language translation of the annexes to the International Preliminary Examination Report under PCT Article 36 (35 U.S.C. 371(c)(5)). 				
Items 11 to 20 below concern document(s) or information included:				
<ol style="list-style-type: none"> <input checked="" type="checkbox"/> An Information Disclosure Statement under 37 CFR 1.97 and 1.98. <input type="checkbox"/> An assignment document for recording. A separate cover sheet in compliance with 37 CFR 3.28 and 3.31 is included. <input checked="" type="checkbox"/> A FIRST preliminary amendment. <input type="checkbox"/> A SECOND or SUBSEQUENT preliminary amendment. <input type="checkbox"/> A substitute specification. <input type="checkbox"/> A change of power of attorney and/or address letter. <input type="checkbox"/> A computer-readable form of the sequence listing in accordance with PCT Rule 13ter.2 and 35 U.S.C. 1.821 - 1.825. <input type="checkbox"/> A second copy of the published international application under 35 U.S.C. 154(d)(4). <input type="checkbox"/> A second copy of the English language translation of the international application under 35 U.S.C. 154(d)(4). <input checked="" type="checkbox"/> Other items or information: 				
Form PCT/IB/306; Form PCT/IB/308; Form PCT/IB/332; Form PCT/IPEA/416; Form PCT/IPEA/409; International Search Report; and 1 Sheet of Drawings with 2 Figures.				



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U.S. APPLICATION NO (if known, see 37 CFR 1.5)	INTERNATIONAL APPLICATION NO.	ATTORNEY'S DOCKET NUMBER	
Unassigned	PCT/FR00/01809	022701-977	
21. <input checked="" type="checkbox"/> The following fees are submitted:		CALCULATIONS	
Basic National Fee (37 CFR 1.492(a)(1)-(5)):			
<p>Neither international preliminary examination fee (37 CFR 1.482) nor international search fee (37 CFR 1.445(a)(2)) paid to USPTO and International Search Report not prepared by the EPO or JPO</p> <p>International preliminary examination fee (37 CFR 1.482) not paid to USPTO but International Search Report prepared by the EPO or JPO</p> <p>International preliminary examination fee (37 CFR 1.482) not paid to USPTO but international search fee (37 CFR 1.445(a)(2)) paid to USPTO</p> <p>International preliminary examination fee (37 CFR 1.482) paid to USPTO but all claims did not satisfy provisions of PCT Article 33(1)-(4)</p> <p>International preliminary examination fee (37 CFR 1.482) paid to USPTO and all claims satisfied provisions of PCT Article 33(1)-(4)</p>		\$1,040.00 (960) \$890.00 (970) \$740.00 (958) \$710.00 (956) \$100.00 (962)	
ENTER APPROPRIATE BASIC FEE AMOUNT =		\$ 890.00	
Surcharge of \$130.00 (154) for furnishing the oath or declaration later than months from the earliest claimed priority date (37 CFR 1.492(e)).		20 <input type="checkbox"/> 30 <input type="checkbox"/>	
Claims	Number Filed	Number Extra	
Total Claims	8 -20 =	X\$18.00 (966)	
Independent Claims	1 -3 =	X\$84.00 (964)	
Multiple dependent claim(s) (if applicable)		+\$280.00 (968)	
TOTAL OF ABOVE CALCULATIONS =		\$ 890.00	
Reduction for 1/2 for filing by small entity, if applicable (see below).		+ \$ -	
SUBTOTAL =		\$ 890.00	
Processing fee of \$130.00 (156) for furnishing the English translation later than months from the earliest claimed priority date (37 CFR 1.492(f)).		20 <input type="checkbox"/> 30 <input type="checkbox"/> +	
TOTAL NATIONAL FEE =		\$ 890.00	
Fee for recording the enclosed assignment (37 CFR 1.21(h)). The assignment must be accompanied by an appropriate cover sheet (37 CFR 3.28, 3.31). \$40.00 (581) per property		+ \$	
TOTAL FEES ENCLOSED =		\$ 890.00	
		Amount to be refunded: \$	
		charged: \$	
a. <input type="checkbox"/> Small entity status is hereby claimed.			
b. <input checked="" type="checkbox"/> A check in the amount of \$ 890.00 to cover the above fees is enclosed.			
c. <input type="checkbox"/> Please charge my Deposit Account No. 02-4800 in the amount of \$ to cover the above fees. A duplicate copy of this sheet is enclosed.			
d. <input checked="" type="checkbox"/> The Commissioner is hereby authorized to charge any additional fees which may be required, or credit any overpayment to Deposit Account No. 02-4800. A duplicate copy of this sheet is enclosed.			
NOTE: Where an appropriate time limit under 37 CFR 1.494 or 1.495 has not been met, a petition to revive (37 CFR 1.137(a) or (b)) must be filed and granted to restore the application to pending status.			
SEND ALL CORRESPONDENCE TO:			
Norman H. Stepno BURNS, DOANE, SWECKER & MATHIS, L.L.P. P.O. Box 1404 Alexandria, Virginia 22313-1404 (703) 836-6620			
SIGNATURE		Teresa Stanek Rea	
NAME		30,427	
REGISTRATION NUMBER		December 21, 2001	
DATE			

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Patent Application of)
Albert FELIX *et al.*) Group Art Unit: Unassigned
Application No.: Unassigned) Examiner: Unassigned
(Corresponds to PCT/FR00/01809))
International Filing Date: 28 JUNE 2000)
For: METHOD FOR MAKING ADIPIC)
ACID)

PRELIMINARY AMENDMENT

Assistant Commissioner for Patents
Washington, D.C. 20231

Sir:

Prior to examination, please amend the above-captioned application as follows:

IN THE CLAIMS:

Kindly amend claims 1-8 as follows.

1. (Amended) Process for the manufacture of adipic acid crystals from adipic acid obtained by crystallization, comprising dispersing the adipic acid crystals collected on conclusion of crystallization in a liquid medium, stirring said liquid medium and separating said crystals from said liquid medium.

2. (Amended) Process according to Claim 1, wherein the liquid medium is water or a water/acetic acid mixture.

3. (Amended) Process according to Claim 1, wherein the temperature of the liquid medium is between 20°C and 70°C.

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4. (Amended) Process according to Claim 1, wherein the concentration by weight of adipic acid in the liquid medium is greater than or equal to 5%.

5. (Amended) Process according to Claim 4, wherein the concentration by weight of adipic acid in the liquid medium is between 5% and 60%.

6. (Amended) Process according to Claim 1, wherein the adipic acid crystals before dispersion have a mean size of between 100 μm and 1000 μm .

7. (Amended) Process according to Claim 1, wherein the crystals separated from the dispersion have a size of between 50 μm and 1000 m.

8. Process according to Claim 1, wherein the liquid medium is cooled before the separation of the treated crystals.

REMARKS

Entry of the foregoing amendments are respectfully requested.

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Should the Examiner have any questions concerning the subject application, a telephone call to the undersigned would be appreciated.

Respectfully submitted,

BURNS, DOANE, SWECKER & MATHIS, L.L.P.

By: _____

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Date: December 21, 2001

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Mark-up of Claims - Page 1 of 1

Attachment to Preliminary Amendment dated December 21, 2001
Mark-up of Claims 1-8

1. (Amended) Process for the manufacture of adipic acid crystals from adipic acid obtained by crystallization, [characterized in that it consists in] comprising dispersing the adipic acid crystals collected on conclusion of crystallization in a liquid medium, [in] stirring [the] said liquid medium and [in then] separating [the] said crystals from [the] said liquid medium.
2. (Amended) Process according to Claim 1, [characterized in that] wherein the liquid medium is water or a water/acetic acid mixture.
3. (Amended) Process according to Claim 1 [or 2], [characterized in that] wherein the temperature of the liquid medium is between 20°C and 70°C.
4. (Amended) Process according to [one of the preceding claims] Claim 1, [characterized in that] wherein the concentration by weight of adipic acid in the liquid medium is greater than or equal to 5%.
5. (Amended) Process according to Claim 4, [characterized in that] wherein the concentration by weight of adipic acid in the liquid medium is between 5% and 60%.
6. (Amended) Process according to [one of the preceding claims] Claim 1, [characterized in that] wherein the adipic acid crystals before dispersion have a mean size of between 100 μm and 1000 μm .
7. (Amended) Process according to [one of the preceding claims] Claim 1, [characterized in that] wherein the crystals separated from the dispersion have a size of between 50 μm and 1000 m.

1990年1月1日，原中国科学院

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Mark-up of Claims 1-8

8. Process according to [one of the preceding claims] Claim 1, [characterized in that] wherein the liquid medium is cooled before the separation of the treated crystals.

11/PPTS

10/018814

JC03 Rec'd PG

21 DEC 2001

1

PROCESS FOR THE MANUFACTURE OF ADIPIC ACID

The present invention relates to a process for the manufacture of adipic acid, more particularly
5 of adipic acid crystals.

It relates more specifically to a process for the treatment of adipic acid crystals obtained on conclusion of crystallization.

Adipic acid is a major intermediate, in
10 particular in the field of polymers and more particularly of polyamide and in the synthesis of polyurethanes.

Adipic acid is generally synthesized by oxidation of a cyclohexanone/cyclohexanol mixture by
15 nitric acid in the presence of oxidation catalysts, such as vanadium and copper.

The adipic acid is recovered and purified by successive crystallization operations.

During these operations, the adipic acid is
20 separated in particular from the other dicarboxylic acids formed, such as glutaric acid or succinic acid.

The adipic acid crystals produced on conclusion of crystallization are generally oblong in shape and have a very uneven surface.

25 Adipic acid is generally stored in large tanks or containers and optionally transported to the site of its use, for example plants for the manufacture of polyamide or of hexamethylenediamine adipate salt.

During this storage and optionally transportation, caking often takes place, that is to say a sticking together of several crystals. This phenomenon is highly damaging as it greatly reduces the 5 flowability of adipic acid during the feeding of the crystals in the plants, for example polymerization plants.

Patent US 5,471,001 provides a specific process for the crystallization of adipic acid with the 10 use of ultrasound. The crystals obtained exhibit better flowability and a weaker property of "caking" during storage and transportation.

One of the aims of the present invention is to provide a process which makes possible the 15 manufacture of adipic acid crystals exhibiting a specific shape and a specific surface condition, in order to obtain good flowability during the charging or discharging or more generally the transportation of these crystals and a decreased property of "caking".

To this end, the invention provides a process 20 for the manufacture of adipic acid crystals which consists in treating the crystals obtained after crystallization according to a process comprising the stages of dispersing the said crystals in a liquid 25 medium, of stirring the said liquid medium for a predetermined period of time, in order to obtain the desired shape and the desired surface condition of the

crystals, and of then separating the said treated crystals from the said liquid medium.

According to another characteristic of the invention, the liquid medium is preferably water or a
5 water/acetic acid mixture in all proportions.

The temperature conditions for carrying out this treatment, in particular during the stage of stirring the liquid medium, are not critical.

However, it is preferable for the solubility
10 of adipic acid in the liquid medium to remain low in
the temperature range chosen, for example at a value of
less than approximately 2 g/l at 20°C. The preferred
temperature range of the invention is between 20°C and
70°C, advantageously between 20°C and 60°C.

15 In some cases, it may be advantageous to cool
the liquid medium to a temperature of less than 20°C,
before separating the crystals from the said liquid
medium.

Likewise, the power of the stirring should be sufficient to prevent excessively large concentration gradients for adipic acid or other compounds present in the medium. However, this power should not be too high, in order to avoid breaking up the crystals.

According to a novel characteristic of the
invention, the concentration by weight of adipic acid
crystals in the liquid medium is greater than 5% (solid
mass/solid mass + liquid mass ratio) and advantageously
between 5% and 60% by weight.

The concentration of crystals can have an effect on the result of the treatment. This is because the greater the number of crystals, the greater the effect of smoothing the surface of the latter. However, 5 an excessively high concentration can be harmful to the process as it will not make it possible to obtain a correct smoothing effect and can result in an agglomeration of crystals to one another.

According to the invention, it is preferable 10 for the adipic acid crystals intended to be dispersed to exhibit a mean size of between 100 µm and 1000 µm, advantageously between 200 and 700 µm.

These crystals can be subjected to a preliminary milling, if their mean size after 15 crystallization is too high.

After treatment according to the process, the crystals have a mean size of between 50 µm and 1000 µm approximately.

However, the mean sizes specified hereinabove 20 are only indicated by way of illustration and correspond to the preferred ranges. The process of the invention can also be applied to crystals with a lower or greater mean size.

According to a preferred embodiment of the 25 invention, the treatment of the crystals is obtained by setting the liquid medium in motion. This setting of the liquid medium in motion can be a stirring of the latter carried out with one or more stirrers exhibiting

varied moving forms conventionally used in the field of the stirring of suspensions.

To improve the effect of the treatment, baffles can be positioned in the reactor comprising the 5 liquid medium.

This setting of the liquid medium in motion can also be obtained by rotating the liquid medium in a device of the centrifuge type.

Finally, the invention generally comprises 10 any means and device capable of setting a liquid in motion in a reactor or a vessel. This is because other plants or devices than those described hereinabove might be used without departing, on that account, from the scope of the invention.

15 Furthermore, the process of the invention makes it possible, if necessary, to wash the adipic acid crystals. Thus, the content of nitric acid is greatly reduced.

The adipic acid crystals treated according to 20 the process of the invention have the form of a pebble with a smooth surface. The pebbles have varied shapes, in particular oblong shapes, exhibiting no sharp ridges.

25 The crystals thus treated exhibit a low tendency to cake. In addition, their shape, without sharp ridges, and their smooth surface make it possible to obtain ready movement of one agglomerate with

respect to another when they are isolated from the liquid medium and dried.

For this reason, the adipic acid crystals obtained by the process of the invention exhibit 5 excellent flowability and a very weak property of caking.

It is therefore possible to store and transport these products for lengthy periods of time and under uncontrolled atmospheric conditions.

10 It is easy to fill the storage and transportation containers, as well as to remove from stock or to feed to reactors.

The invention will be better illustrated in the light of the examples hereinbelow, given solely by 15 way of indication, and with reference to the appended figures, in which:

- Figure 1 represents a view taken with a scanning electron microscope with a magnification of 20 of a sample of crystals of adipic acid untreated by the 20 process of the invention, and

- Figure 2 represents a view taken with a scanning electron microscope with a magnification of 20 of a sample of the crystals represented in Figure 1 after treatment by the process of the invention.

25 Adipic acid crystals obtained by crystallization from an aqueous adipic acid solution have a mean size of 600 µm. The form of these crystals is illustrated in Figure 1. These crystals form lumps

with an oblong shape exhibiting a very uneven surface, comprising small grains stuck to or agglomerated at the surface and numerous cracks or cavities. 200 g of crystals are dispersed in 330 g of water present in a 5 vessel equipped with a stirrer. The concentration of the crystals in the liquid medium is 40% by weight.

The mixture is kept stirred for one hour at a temperature of 25°C.

After filtering and drying in a fluidized bed 10 at 100°C for one hour, the adipic acid crystals obtained exhibit a mean size of 600 µm.

The appearance of these crystals, illustrated by Figure 2, clearly shows the effect of the process of the invention. This is because the crystals still have 15 the form of an oblong pebble but their surface is smooth with few adherent particles.

After storing the crystals in a conventional container for several days in a normal atmosphere, feeding them to a reactor did not present any problems. 20 No agglomeration or caking was observed.

CLAIMS

1. Process for the manufacture of adipic acid crystals from adipic acid obtained by crystallization, characterized in that it consists in dispersing the adipic acid crystals collected on conclusion of crystallization in a liquid medium, in stirring the said liquid medium and in then separating the said crystals from the said liquid medium.
- 10 2. Process according to Claim 1, characterized in that the liquid medium is water or a water/acetic acid mixture.
3. Process according to Claim 1 or 2, characterized in that the temperature of the liquid medium is between 20°C and 70°C.
- 15 4. Process according to one of the preceding claims, characterized in that the concentration by weight of adipic acid in the liquid medium is greater than or equal to 5%.
- 20 5. Process according to Claim 4, characterized in that the concentration by weight of adipic acid in the liquid medium is between 5% and 60%.
6. Process according to one of the preceding claims, characterized in that the adipic acid 25 crystals before dispersion have a mean size of between 100 µm and 1000 µm.
7. Process according to one of the preceding claims, characterized in that the crystals

separated from the dispersion have a size of between 50 μm and 1000 μm .

8. Process according to one of the preceding claims, characterized in that the liquid medium is cooled before the separation of the treated crystals.

PROCESS FOR THE MANUFACTURE OF ADIPIC ACID

The present invention relates to a process for the manufacture of adipic acid, more particularly of adipic acid crystals.

It relates more specifically to a process for the treatment of the adipic acid crystals obtained on conclusion of crystallization, which consists in dispersing the adipic acid crystals collected on conclusion of crystallization in a liquid medium, in stirring the said liquid medium and in then separating the said crystals from the said liquid medium and optionally drying them.

The form of the crystals is modified in order to obtain pebbles with a substantially smooth surface.

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En ce qui concerne les codes à deux lettres et autres abréviations, se référer aux "Notes explicatives relatives aux codes et abréviations" figurant au début de chaque numéro ordinaire de la Gazette du PCT.

(72) Inventeurs; et

(75) Inventeurs/Déposants (pour US seulement): FELIX, Albert [FR/FR]; 79, rue du 8 Mai 1945, F-69100 Villeurbanne (FR). ROQUES, Yves [FR/FR]; 9, rue d'Hanoï, F-69100 Villeurbanne (FR).

(54) Title: METHOD FOR MAKING ADIPIC ACID

A1 **(54) Titre:** PROCEDE DE FABRICATION D'ACIDE ADIPIQUE

(57) Abstract: The invention concerns a method for making adipic acid, more particularly adipic acid crystals. More precisely, it concerns a method for treating adipic acid crystals derived from crystallisation which consists in dispersing the adipic acid crystals collected at the output of crystallisation in a liquid medium, stirring said liquid medium, then separating said crystals and optionally drying them. The shape of the crystals is modified to obtain pebbles with substantially smooth surface.

(57) Abrégé: La présente invention concerne un procédé de fabrication d'acide adipique, plus particulièrement de cristaux d'acide adipique. Elle est relative plus concrètement à un procédé de traitement des cristaux d'acide adipique obtenus en sortie de cristallisation consistant à disperser les cristaux d'acide adipique recueillis en sortie de cristallisation dans un milieu liquide, en agitant ledit milieu liquide puis à séparer lesdits cristaux dudit milieu liquide et éventuellement les sécher. La forme des cristaux est modifiée pour obtenir des galets de surface sensiblement lisse.

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1/1

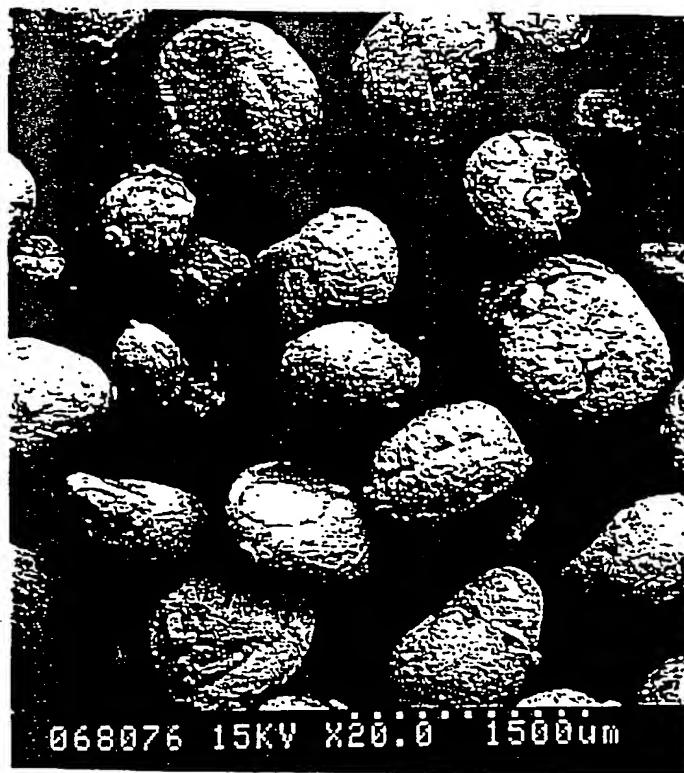


Fig. 1

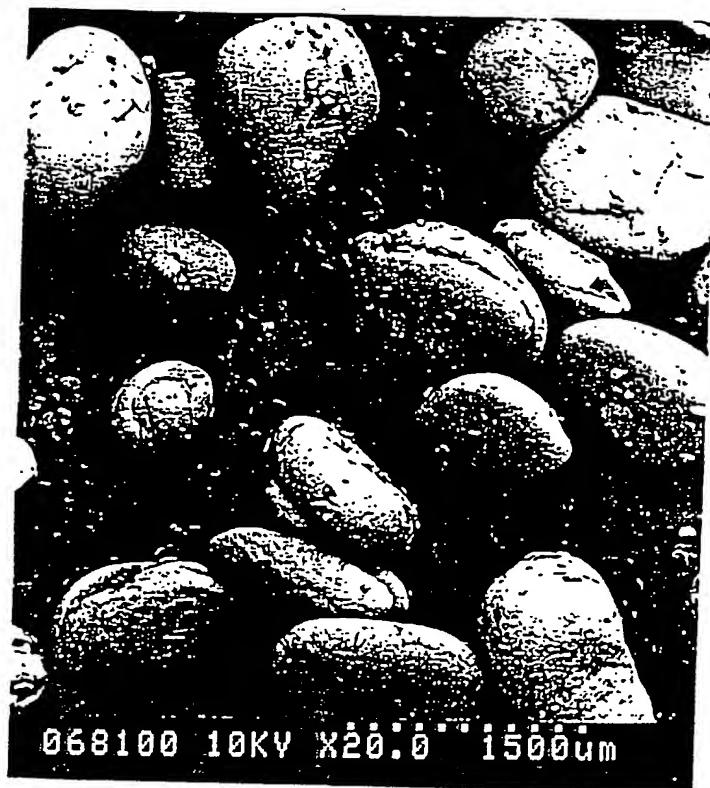


Fig. 2

022701-977
Attorney's Docket No.

**COMBINED DECLARATION AND POWER OF ATTORNEY
FOR UTILITY OR DESIGN PATENT APPLICATION**

As a below named inventor, I hereby declare that:

My residence, post office address and citizenship are as stated below next to my name;

I believe I am the original, first and sole inventor (if only one name is listed below) or an original, first and joint inventor (if plural names are listed below) of the subject matter which is claimed and for which a patent is sought on the invention entitled:

METHOD FOR MAKING ADIPIC ACID

the specification of which (check only one item below):

- is attached hereto.
- was filed as United States application
Number _____ on _____
and was amended _____ on _____ (if applicable).
- was filed as PCT international application
Number PCT/FR00/01809 on 28 JUNE 2000
and was amended _____ on 21 DECEMBER 2001 (if applicable).

I hereby state that I have reviewed and understand the contents of the above-identified specification, including the claims, as amended by any amendment referred to above.

I acknowledge the duty to disclose to the Office all information known to me to be material to patentability as defined in Title 37, Code of Federal Regulations, §1.56.

I hereby claim foreign priority benefits under Title 35, United States Code, §§119 (a)-(d), 172 or 365 of any foreign application(s) for patent or inventor's certificate or of any PCT international application(s) designating at least one country other than the United States of America listed below and have also identified below any foreign application(s) for patent or inventor's certificate or any PCT international application(s) designating at least one country other than the United States of America filed by me on the same subject matter having a filing date before that of the application(s) of which priority is claimed:

PRIOR FOREIGN/PCT APPLICATION(S) AND ANY PRIORITY CLAIMS UNDER 35 U.S.C. §§119(a)-(d), 172 or 365:			
COUNTRY (if PCT, indicate "PCT")	APPLICATION NUMBER	DATE OF FILING (day, month, year)	PRIORITY CLAIMED UNDER 35 U.S.C. §§119, 172 or 365
FRANCE	99/08591	29 JUNE 1999	X Yes No
			Yes No
			Yes No
			Yes No
			Yes No

Combined Declaration and Power of Attorney
for Utility or Design Patent Application
Attorney's Docket No. 022701-977
Page 2 of 2

I hereby appoint the following attorneys and agent(s) to prosecute said application and to transact all business in the Patent and Trademark Office connected therewith and to file, prosecute and to transact all business in connection with international applications directed to said invention:

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21839

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I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

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Signature	
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Citizenship	FRANCE
Mailing Address	79, rue du 8 Mai 1945, F-69100 Villeurbanne, FRANCE
City, State, ZIP, Country	F-69100 Villeurbanne, FRANCE
FULL NAME SECOND INVENTOR, IF ANY	Yves ROQUES
Signature	
Date	10/04/2002
Residence (City, State, Country)	Villeurbanne, FRANCE FRX
Citizenship	FRANCE
Mailing Address	9, rue d'Hanoï, F-69100 Villeurbanne, FRANCE
City, State, ZIP, Country	F-69100 Villeurbanne, FRANCE